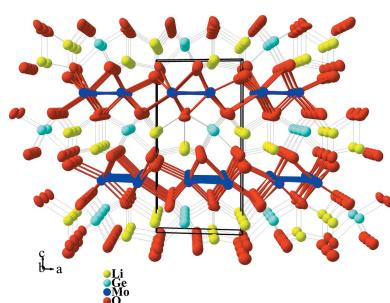




Received 3 June 2016

Accepted 15 June 2016

Edited by M. Weil, Vienna University of
Technology, Austria**Keywords:** crystal structure; reduced molybdenum oxide; triangular Mo_3 cluster; lithium; germanium.**CCDC reference:** 1485831**Supporting information:** this article has supporting information at journals.iucr.org/e

OPEN ACCESS

$\text{Li}_2\text{GeMo}_3\text{O}_8$: a novel reduced molybdenum oxide containing Mo_3O_{13} cluster units

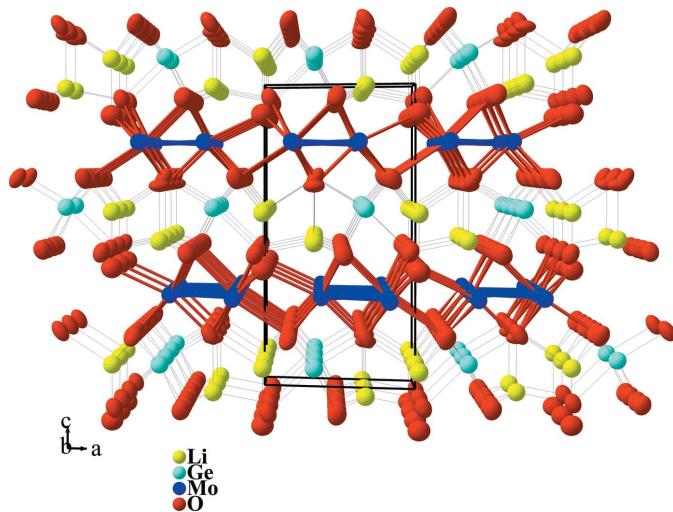
Philippe Gall and Patrick Gougeon*

Institut des Sciences, Chimiques de Rennes, UMR 6226 CNRS – INSA Rennes – Université de Rennes 1, Avenue du Général Leclerc, 35042 Rennes Cedex, France. *Correspondence e-mail: patrick.gougeon@univ-rennes1.fr

The crystal structure of the title compound, dilithium germanium trimolybdenum octaoxide, consists of distorted hexagonal-close-packed oxygen layers with stacking sequence *ABAC* along [001] that are held together by alternating lithium–germanium and molybdenum layers. The two Li^+ and Ge^{4+} ions all have site symmetry *3m*. and occupy, respectively, tetrahedral and octahedral sites in the ratio 2:1. The Mo atom has a formal oxidation state of +3.3 and occupies an octahedral site (site symmetry *m*) and forms strongly bonded triangular cluster units [$\text{Mo}–\text{Mo}$ distance = 2.4728 (8) Å] involving three MoO_6 octahedra that are each shared along two edges, constituting an Mo_3O_{13} unit.

1. Chemical context

Reduced molybdenum oxides containing the Mo_3O_{13} cluster unit crystallize either in the hexagonal space group type $P6_3mc$ ($a \sim 5.7\text{--}5.8$ Å, $c \sim 10.0\text{--}10.2$ Å) or in the trigonal space group types $P3m1$ ($a \sim 5.7\text{--}5.8$ Å, $c \sim 4.9\text{--}5.3$ Å) or $R\bar{3}m$ ($a \sim 5.8\text{--}5.9$ Å, $c \sim 30.0\text{--}30.1$ Å). Representatives of the first family are the ternary compounds $M_2\text{Mo}_3\text{O}_8$ (McCarroll *et al.*, 1957) where M is a divalent metal such as Mg, Zn, Fe, Co, Ni, Zn and Cd as well as the quaternary compounds $\text{ScZnMo}_3\text{O}_8$ and $\text{Li}_2MMo_3\text{O}_8$ ($M = \text{Sn, In}$) (Gall *et al.*, 2013*a,b*). The LiRMo_3O_8 series ($R = \text{Sc, Y, In, Sm, Gd, Tb, Dy, Ho, Er and Yb}$) (DeBenedittis & Katz, 1965; McCarroll, 1977) crystallize in the $P3m1$ space group and finally, $\text{LiZn}_2\text{Mo}_3\text{O}_8$ and $\text{Zn}_3\text{Mo}_3\text{O}_8$ (Torardi & McCarley, 1985) crystallize in space group $R\bar{3}m$. The crystal structures of all these compounds consist of distorted hexagonal-close-packed oxygen layers with stacking sequences *ABAC*, *ABAB* and *ABC* for compounds crystallizing in the space groups $P6_3mc$, $P3m1$ and $R\bar{3}m$, respectively. The oxygen layers are separated by alternating mixed-metal atom (Li, M , or R) layers and molybdenum layers. The metal atoms occupy both tetrahedral and octahedral sites in a ratio of 1:1 ($M_2\text{Mo}_3\text{O}_8$ and LiRMo_3O_8) or 2:1 ($\text{LiZn}_2\text{Mo}_3\text{O}_8$ and $\text{Zn}_3\text{Mo}_3\text{O}_8$) between two adjacent oxygen layers. The molybdenum atoms occupy three quarters of the octahedral sites and form strongly bonded triangular cluster units involving three MoO_6 octahedra that are each shared along two edges, the whole constituting an Mo_3O_{13} unit. The $\text{Mo}–\text{Mo}$ bonds within the trinuclear cluster units range from about 2.5 to 2.6 Å, and the number of electrons available for $\text{Mo}–\text{Mo}$ bonding is six in $M_2\text{Mo}_3\text{O}_8$ and LiRMo_3O_8 , seven in $\text{LiZn}_2\text{Mo}_3\text{O}_8$ and $\text{Li}_2\text{InMo}_3\text{O}_8$, and eight in $\text{Zn}_3\text{Mo}_3\text{O}_8$ and $\text{Li}_2\text{SnMo}_3\text{O}_8$. The energy-level diagram deduced from LCAO-MO calculations on the Mo_3O_{13} unit shows three bonding orbitals ($a1$ and e), a non-bonding level ($a1$), and five anti-

**Figure 1**

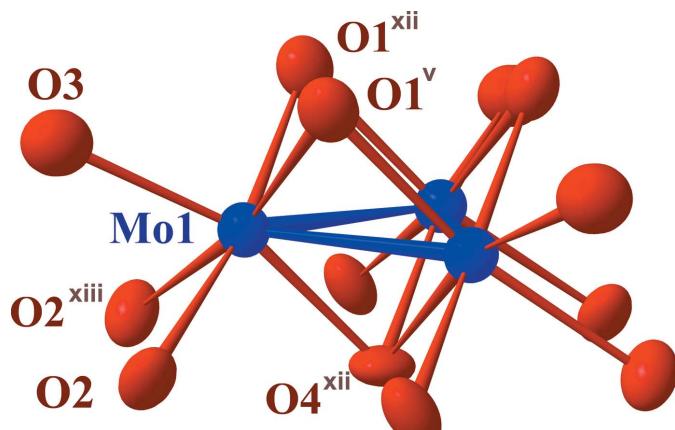
View of the crystal structure of $\text{Li}_2\text{GeMo}_3\text{O}_8$ in a projection approximately along [010]. Displacement ellipsoids are drawn at the 97% probability level.

bonding orbitals ($2e$ and a_2) (Cotton, 1964). This explains why the compounds with seven electrons per Mo_3 cluster unit are paramagnetic with moments corresponding to one unpaired electron per Mo_3 cluster unit, and those with six and eight electrons per Mo_3 show temperature-independent paramagnetism.

We present here the crystal structure of the new quaternary compound $\text{Li}_2\text{GeMo}_3\text{O}_8$ in which the Mo_3 cluster unit has eight electrons available for bonding.

2. Structural commentary

$\text{Li}_2\text{GeMo}_3\text{O}_8$ is isotopic with the $\text{Li}_2\text{MMo}_3\text{O}_8$ ($M = \text{Sn}, \text{In}$) compounds (Gall *et al.*, 2013*a,b*). Its crystal structure consists of distorted hexagonal-close-packed oxygen layers with stacking sequence *ABAC* along [001] that are held together by

**Figure 2**

The Mo_3O_{13} cluster unit with its numbering scheme, with ellipsoids drawn at the 97% probability level. [Symmetry codes: (v) $y + 1, -x + y + 2, z - \frac{1}{2}$; (xii) $-x + 3, -y + 2, z - \frac{1}{2}$; (xiii) $-y + 2, x - y + 1, z$; (xiv) $-x + y + 2, -x + 3, z$.]

Table 1
Selected bond lengths (\AA).

$\text{Li1}-\text{O3}$	1.84 (2)	$\text{Ge1}-\text{O1}^{\text{x}}$	2.016 (5)
$\text{Li1}-\text{O2}^{\text{i}}$	2.012 (13)	$\text{Ge1}-\text{O1}^{\text{x}}$	2.016 (5)
$\text{Li1}-\text{O2}^{\text{ii}}$	2.012 (13)	$\text{Ge1}-\text{O1}^{\text{x}}$	2.016 (4)
$\text{Li1}-\text{O2}^{\text{iii}}$	2.012 (13)	$\text{Mo1}-\text{O4}^{\text{xiii}}$	2.004 (6)
$\text{Li2}-\text{O4}$	1.78 (3)	$\text{Mo1}-\text{O1}^{\text{xii}}$	2.039 (4)
$\text{Li2}-\text{O1}^{\text{iv}}$	1.892 (6)	$\text{Mo1}-\text{O1}^{\text{v}}$	2.039 (4)
$\text{Li2}-\text{O1}^{\text{v}}$	1.892 (6)	$\text{Mo1}-\text{O3}$	2.076 (3)
$\text{Li2}-\text{O1}^{\text{vi}}$	1.892 (6)	$\text{Mo1}-\text{O2}$	2.146 (3)
$\text{Ge1}-\text{O2}$	1.883 (5)	$\text{Mo1}-\text{O2}^{\text{xiii}}$	2.146 (3)
$\text{Ge1}-\text{O2}^{\text{vii}}$	1.883 (5)	$\text{Mo1}-\text{Mo1}^{\text{xiv}}$	2.4728 (8)
$\text{Ge1}-\text{O2}^{\text{viii}}$	1.883 (5)		

Symmetry codes: (i) $x - y + 1, x, z + \frac{1}{2}$; (ii) $-x + 2, -y + 2, z + \frac{1}{2}$; (iii) $y, -x + y + 1, z + \frac{1}{2}$; (iv) $x - y, x - 1, z - \frac{1}{2}$; (v) $y + 1, -x + y + 2, z - \frac{1}{2}$; (vi) $-x + 3, -y + 1, z - \frac{1}{2}$; (vii) $-x + y + 2, -x + 2, z$; (viii) $-y + 2, x - y, z$; (ix) $-x + y + 2, -x + 2, z - 1$; (x) $-y + 2, x - y, z - 1$; (xi) $x, y, z - 1$; (xii) $-x + 3, -y + 2, z - \frac{1}{2}$; (xiii) $-y + 2, x - y + 1, z$; (xiv) $-x + y + 2, -x + 3, z$.

alternating lithium–germanium and molybdenum layers (Fig. 1). The Li^+ and Ge^{4+} ions occupy, respectively, tetrahedral and octahedral sites in the ratio 2:1. The Mo atoms occupy octahedral sites and form strongly bonded triangular cluster units involving three Mo_6 octahedra that are each shared along two edges, constituting an Mo_3O_{13} unit (Fig. 2). The Mo–Mo distance within the Mo_3 triangle is 2.4728 (8) \AA compared to 2.5036 (7) and 2.5455 (4) \AA found in the tin and indium analogues, respectively. The Mo–O distances range from 2.004 (6) to 2.146 (3) \AA (Table 1) while in $\text{Li}_2\text{InMo}_3\text{O}_8$ they range from 2.0212 (17) to 2.1241 (16) \AA and in $\text{Li}_2\text{SnMo}_3\text{O}_8$ from 2.020 (6) to 2.122 (3) \AA . The Li–O distances in the title structure range from 1.78 (2) to 2.012 (13) \AA with average distances of 1.97 and 1.86 \AA for the Li1 and Li2 sites, respectively. Both Li sites have site symmetry $3m$. For the Ge site, likewise with site symmetry $3m$, the Ge–O distances are 3×1.883 (5) and 3×2.016 (5) \AA . The average distance of 1.95 \AA is close to the value of 1.92 \AA calculated from the sum of the ionic radii of O^{2-} and Ge^{4+} in octahedral coordination according to Shannon & Prewitt (1969). The oxidation state of +4 for the Ge atoms was also confirmed from the Ge–O bond lengths by using the relationship of Brown & Wu (1976) $\{s = [d(\text{Ge}–\text{O})/1.746]^{-0.05}\}$ which leads to a value of +3.5 (1). The latter relationship applied to Mo–O bonds $\{s = [d(\text{Mo}–\text{O})/1.882]^{-6}\}$ yield an oxidation state of +3.38 for the Mo atom, and thus 7.86 electrons per Mo_3 cluster unit, close to the expected value of 8. This is consistent with the chemical composition $\text{Li}_2^+\text{Ge}^{4+}\text{Mo}_3^{3.33+}\text{O}_8^{2-}$.

3. Database survey

The $M_2\text{Mo}_3\text{O}_8$ ($M = \text{Mg}, \text{Zn}, \text{Fe}, \text{Co}, \text{Ni}, \text{Zn}$ and Cd) compounds containing triangular Mo_3 clusters were first synthesized by McCarroll *et al.* (1957). They presented the results of a structure determination on $\text{Zn}_2\text{Mo}_3\text{O}_8$ from photographic data ($R = 0.118$). Later, a refinement of the structure was accomplished by Ansell & Katz (1966) with an R factor of 0.069. Among the above compounds, it is interesting to note that $\text{Fe}_2\text{Mo}_3\text{O}_8$ is a mineral known as kamiokite (Kanazawa & Sasaki, 1986). Later, DeBenedittis & Katz (1965) reported the

Table 2
Experimental details.

Crystal data	
Chemical formula	$\text{Li}_2\text{GeMo}_3\text{O}_8$
M_r	502.29
Crystal system, space group	Hexagonal, $P6_3mc$
Temperature (K)	293
a, c (Å)	5.7268 (3), 9.9841 (6)
V (Å ³)	283.57 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	11.74
Crystal size (mm)	0.21 × 0.13 × 0.07
Data collection	
Diffractometer	Nonius KappaCCD
Absorption correction	Analytical (de Meulenaer & Tompa, 1965)
T_{\min}, T_{\max}	0.048, 0.157
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4457, 522, 501
R_{int}	0.063
(sin θ/λ) _{max} (Å ⁻¹)	0.807
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.030, 0.076, 1.10
No. of reflections	522
No. of parameters	31
No. of restraints	1
$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e Å ⁻³)	1.43, -1.33
Absolute structure	Flack (1983), 247 Friedel pairs
Absolute structure parameter	0.01 (3)

Computer programs: *COLLECT* (Nonius, 1998), *EVALCCD* (Duisenberg *et al.*, 2003), *SIR97* (Altomare *et al.*, 1999), *SHELXL97* (Sheldrick, 2008) and *DIAMOND* (Bergerhoff, 1996).

existence of the $\text{Li}_RMo_3\text{O}_8$ ($R = \text{Sc}$ and Y) compounds. Subsequently, McCarroll (1977) obtained isotypic compounds with $R = \text{In}$, Sm , Gd , Tb , Dy , Ho , Er , and Yb . In 1985, Torardi & McCarley (1985) described the new Mo_3 cluster compounds $\text{LiZn}_2\text{Mo}_3\text{O}_8$, $\text{Zn}_3\text{Mo}_3\text{O}_8$ and $\text{ScZnMo}_3\text{O}_8$ and, in 2013, Gall *et al.* (2013a,b), the quaternary compounds $\text{Li}_2MMo_3\text{O}_8$ ($M = \text{Sn}$ and In).

4. Synthesis and crystallization

Single crystals of $\text{Li}_2\text{GeMo}_3\text{O}_8$ were obtained by heating a mixture of Li_2MoO_4 , O_2 , MoO_3 and Mo with the nominal

composition $\text{Li}_2\text{GeMo}_3\text{O}_{12}$ at 1923 K for 72 h in a molybdenum crucible sealed under low argon pressure using an arc-welding system. The molybdate Li_2MoO_4 was synthesized by heating an equimolar ratio of MoO_3 (CERAC 99.95%) and Li_2CO_3 (CERAC 99.9%) in an alumina vessel at 873 K in air over 12 h. Before use, the Mo powder was heated under a hydrogen flow at 1273 K for 6 h. The composition of the final crystals thus obtained was determined after a complete X-ray structural study on one of them.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All atoms were refined with anisotropic displacement parameters, except for the Li atoms, which were refined isotropically.

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Ansell, G. B. & Katz, L. (1966). *Acta Cryst.* **21**, 482–485.
- Bergerhoff, G. (1996). *DIAMOND*. University of Bonn, Germany.
- Brown, I. D. & Wu, K. K. (1976). *Acta Cryst.* **B32**, 1957–1959.
- Cotton, F. A. (1964). *Inorg. Chem.* **3**, 1217–1220.
- DeBenedittis, J. & Katz, L. (1965). *Inorg. Chem.* **4**, 1836–1839.
- Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Gall, P., Al Rahal Al Orabi, R., Guizouarn, T., Cuny, J., Fontaine, B., Gautier, R. & Gougeon, P. (2013a). *J. Solid State Chem.* **201**, 312–316.
- Gall, P., Al Rahal Al Orabi, R., Guizouarn, T. & Gougeon, P. (2013b). *J. Solid State Chem.* **208**, 99–102.
- Kanazawa, Y. & Sasaki, A. (1986). *Acta Cryst.* **C42**, 9–11.
- McCarroll, W. H. (1977). *Inorg. Chem.* **16**, 3351–3353.
- McCarroll, W. H., Katz, L. & Ward, R. (1957). *J. Am. Chem. Soc.* **79**, 5410–5414.
- Meulenaer, J. de & Tompa, H. (1965). *Acta Cryst.* **19**, 1014–1018.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Shannon, R. D. & Prewitt, C. T. (1969). *Acta Cryst.* **B25**, 925–946.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Torardi, C. C. & McCarley, R. E. (1985). *Inorg. Chem.* **24**, 476–481.

supporting information

Acta Cryst. (2016). E72, 995-997 [doi:10.1107/S2056989016009750]

Li₂GeMo₃O₈: a novel reduced molybdenum oxide containing Mo₃O₁₃ cluster units

Philippe Gall and Patrick Gougeon

Computing details

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT* (Nonius, 1998); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

Dilithium germanium trimolybdenum octaoxide

Crystal data

Li₂GeMo₃O₈
 $M_r = 502.29$
 Hexagonal, $P\bar{6}_3mc$
 Hall symbol: P 6c -2c
 $a = 5.7268 (3)$ Å
 $c = 9.9841 (6)$ Å
 $V = 283.57 (3)$ Å³
 $Z = 2$
 $F(000) = 456$

$D_x = 5.883$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 4457 reflections
 $\theta = 4.1\text{--}35.0^\circ$
 $\mu = 11.74$ mm⁻¹
 $T = 293$ K
 Irregular block, black
 $0.21 \times 0.13 \times 0.07$ mm

Data collection

Nonius KappaCCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ scans ($\kappa = 0$) + additional ω scans
 Absorption correction: analytical
 (de Meulenaar & Tompa, 1965)
 $T_{\min} = 0.048$, $T_{\max} = 0.157$

4457 measured reflections
 522 independent reflections
 501 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$
 $\theta_{\max} = 35.0^\circ$, $\theta_{\min} = 4.1^\circ$
 $h = -7 \rightarrow 9$
 $k = -9 \rightarrow 9$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.076$
 $S = 1.10$
 522 reflections
 31 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 $w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.3716P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.43$ e Å⁻³
 $\Delta\rho_{\min} = -1.33$ e Å⁻³
 Absolute structure: Flack (1983), 247 Friedel
 pairs
 Absolute structure parameter: 0.01 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F, with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Li1	1.0000	1.0000	1.581 (2)	0.014 (3)*
Li2	1.3333	0.6667	1.490 (2)	0.014 (3)*
Ge1	1.3333	0.6667	1.09391 (9)	0.0076 (2)
Mo1	1.37881 (9)	1.18940 (4)	1.30891 (9)	0.00660 (13)
O1	1.4795 (4)	0.5205 (4)	1.9536 (5)	0.0081 (8)
O2	1.1704 (6)	0.8296 (6)	1.1906 (5)	0.0081 (8)
O3	1.0000	1.0000	1.3974 (7)	0.0110 (14)
O4	1.3333	0.6667	1.6680 (8)	0.0080 (13)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ge1	0.0076 (3)	0.0076 (3)	0.0076 (4)	0.00381 (14)	0.000	0.000
Mo1	0.00622 (19)	0.00662 (16)	0.00683 (19)	0.00311 (10)	-0.00015 (18)	-0.00007 (9)
O1	0.0079 (13)	0.0079 (13)	0.0094 (18)	0.0045 (14)	0.0007 (8)	-0.0007 (8)
O2	0.0071 (12)	0.0071 (12)	0.010 (2)	0.0034 (13)	0.0017 (9)	-0.0017 (9)
O3	0.012 (2)	0.012 (2)	0.010 (3)	0.0058 (10)	0.000	0.000
O4	0.0102 (19)	0.0102 (19)	0.004 (3)	0.0051 (9)	0.000	0.000

Geometric parameters (\AA , ^\circ)

Li1—O3	1.84 (2)	Ge1—O1 ^{xv}	2.016 (5)
Li1—O2 ⁱ	2.012 (13)	Ge1—O1 ^{xvi}	2.016 (4)
Li1—O2 ⁱⁱ	2.012 (13)	Mo1—O4 ^{xvii}	2.004 (6)
Li1—O2 ⁱⁱⁱ	2.012 (13)	Mo1—O1 ^{xviii}	2.039 (4)
Li1—Mo1 ⁱ	2.949 (17)	Mo1—O1 ^{viii}	2.039 (4)
Li1—Mo1 ⁱⁱⁱ	2.949 (17)	Mo1—O3	2.076 (3)
Li1—Mo1 ⁱⁱ	2.949 (17)	Mo1—O2	2.146 (3)
Li1—Mo1 ^{iv}	3.305 (18)	Mo1—O2 ^v	2.146 (3)
Li1—Mo1 ^v	3.305 (18)	Mo1—Mo1 ^{xviii}	2.4728 (8)
Li1—Mo1	3.305 (18)	Mo1—Mo1 ^{xix}	2.4728 (8)
Li1—Li2	3.430 (9)	Mo1—Li1 ^{xx}	2.949 (17)
Li1—Li2 ^{vi}	3.430 (9)	O1—Li2 ^{xxi}	1.892 (6)
Li2—O4	1.78 (3)	O1—Ge1 ^{xxii}	2.016 (4)
Li2—O1 ^{vii}	1.892 (6)	O1—Mo1 ^{xxiii}	2.039 (4)
Li2—O1 ^{viii}	1.892 (6)	O1—Mo1 ^{xxiv}	2.039 (4)

Li2—O1 ^{ix}	1.892 (6)	O2—Li1 ^{xx}	2.012 (13)
Li2—Li1 ^x	3.430 (9)	O2—Mo1 ^{iv}	2.146 (3)
Li2—Li1 ^{xi}	3.430 (9)	O3—Mo1 ^{iv}	2.076 (3)
Ge1—O2	1.883 (5)	O3—Mo1 ^v	2.076 (3)
Ge1—O2 ^{xii}	1.883 (5)	O4—Mo1 ⁱⁱⁱ	2.004 (5)
Ge1—O2 ^{xiii}	1.883 (5)	O4—Mo1 ^{xxiv}	2.004 (5)
Ge1—O1 ^{xiv}	2.016 (5)	O4—Mo1 ^{xxiii}	2.004 (5)
O3—Li1—O2 ⁱ	122.9 (5)	Li1 ^x —Li2—Li1 ^{xi}	113.2 (5)
O3—Li1—O2 ⁱⁱ	122.9 (5)	O2—Ge1—O2 ^{xii}	96.1 (2)
O2 ⁱ —Li1—O2 ⁱⁱ	93.3 (7)	O2—Ge1—O2 ^{xiii}	96.1 (2)
O3—Li1—O2 ⁱⁱⁱ	122.9 (5)	O2 ^{xii} —Ge1—O2 ^{xiii}	96.1 (2)
O2 ⁱ —Li1—O2 ⁱⁱⁱ	93.3 (7)	O2—Ge1—O1 ^{xiv}	92.73 (17)
O2 ⁱⁱ —Li1—O2 ⁱⁱⁱ	93.3 (7)	O2 ^{xii} —Ge1—O1 ^{xiv}	166.8 (2)
O3—Li1—Mo1 ⁱ	140.4 (3)	O2 ^{xiii} —Ge1—O1 ^{xiv}	92.73 (18)
O2 ⁱ —Li1—Mo1 ⁱ	46.7 (4)	O2—Ge1—O1 ^{xv}	92.73 (17)
O2 ⁱⁱ —Li1—Mo1 ⁱ	46.7 (4)	O2 ^{xii} —Ge1—O1 ^{xv}	92.73 (17)
O2 ⁱⁱⁱ —Li1—Mo1 ⁱ	96.7 (8)	O2 ^{xiii} —Ge1—O1 ^{xv}	166.8 (2)
O3—Li1—Mo1 ⁱⁱⁱ	140.4 (3)	O1 ^{xiv} —Ge1—O1 ^{xv}	77.0 (2)
O2 ⁱ —Li1—Mo1 ⁱⁱⁱ	46.7 (4)	O2—Ge1—O1 ^{xvi}	166.8 (2)
O2 ⁱⁱ —Li1—Mo1 ⁱⁱⁱ	96.7 (8)	O2 ^{xii} —Ge1—O1 ^{xvi}	92.73 (17)
O2 ⁱⁱⁱ —Li1—Mo1 ⁱⁱⁱ	46.7 (4)	O2 ^{xiii} —Ge1—O1 ^{xvi}	92.73 (17)
Mo1 ⁱ —Li1—Mo1 ⁱⁱⁱ	67.0 (4)	O1 ^{xiv} —Ge1—O1 ^{xvi}	77.0 (2)
O3—Li1—Mo1 ⁱⁱ	140.4 (3)	O1 ^{xv} —Ge1—O1 ^{xvi}	77.0 (2)
O2 ⁱ —Li1—Mo1 ⁱⁱ	96.7 (8)	O4 ^{xvii} —Mo1—O1 ^{xvii}	104.58 (15)
O2 ⁱⁱ —Li1—Mo1 ⁱⁱ	46.7 (4)	O4 ^{xvii} —Mo1—O1 ^{viii}	104.58 (15)
O2 ⁱⁱⁱ —Li1—Mo1 ⁱⁱ	46.7 (4)	O1 ^{xvii} —Mo1—O1 ^{viii}	76.0 (2)
Mo1 ⁱ —Li1—Mo1 ⁱⁱ	67.0 (4)	O4 ^{xvii} —Mo1—O3	160.6 (3)
Mo1 ⁱⁱⁱ —Li1—Mo1 ⁱⁱ	67.0 (4)	O1 ^{xvii} —Mo1—O3	90.60 (16)
O3—Li1—Mo1 ^{iv}	34.6 (2)	O1 ^{viii} —Mo1—O3	90.60 (16)
O2 ⁱ —Li1—Mo1 ^{iv}	133.2 (4)	O4 ^{xvii} —Mo1—O2	87.53 (16)
O2 ⁱⁱ —Li1—Mo1 ^{iv}	133.2 (4)	O1 ^{xvii} —Mo1—O2	167.40 (14)
O2 ⁱⁱⁱ —Li1—Mo1 ^{iv}	88.2 (3)	O1 ^{viii} —Mo1—O2	97.8 (2)
Mo1 ⁱ —Li1—Mo1 ^{iv}	175.1 (5)	O3—Mo1—O2	78.36 (18)
Mo1 ⁱⁱⁱ —Li1—Mo1 ^{iv}	116.94 (6)	O4 ^{xvii} —Mo1—O2 ^v	87.53 (16)
Mo1 ⁱⁱ —Li1—Mo1 ^{iv}	116.94 (6)	O1 ^{xvii} —Mo1—O2 ^v	97.8 (2)
O3—Li1—Mo1 ^v	34.6 (2)	O1 ^{viii} —Mo1—O2 ^v	167.40 (14)
O2 ⁱ —Li1—Mo1 ^v	133.2 (4)	O3—Mo1—O2 ^v	78.36 (18)
O2 ⁱⁱ —Li1—Mo1 ^v	88.2 (3)	O2—Mo1—O2 ^v	86.0 (3)
O2 ⁱⁱⁱ —Li1—Mo1 ^v	133.2 (4)	O4 ^{xvii} —Mo1—Mo1 ^{xviii}	51.91 (12)
Mo1 ⁱ —Li1—Mo1 ^v	116.94 (6)	O1 ^{xvii} —Mo1—Mo1 ^{xviii}	52.67 (9)
Mo1 ⁱⁱⁱ —Li1—Mo1 ^v	175.1 (5)	O1 ^{viii} —Mo1—Mo1 ^{xviii}	90.54 (10)
Mo1 ⁱⁱ —Li1—Mo1 ^v	116.94 (6)	O3—Mo1—Mo1 ^{xviii}	141.60 (11)
Mo1 ^{iv} —Li1—Mo1 ^v	59.0 (4)	O2—Mo1—Mo1 ^{xviii}	139.30 (11)
O3—Li1—Mo1	34.6 (2)	O2 ^v —Mo1—Mo1 ^{xviii}	94.37 (14)
O2 ⁱ —Li1—Mo1	88.2 (3)	O4 ^{xvii} —Mo1—Mo1 ^{xix}	51.91 (12)
O2 ⁱⁱ —Li1—Mo1	133.2 (4)	O1 ^{xvii} —Mo1—Mo1 ^{xix}	90.54 (9)
O2 ⁱⁱⁱ —Li1—Mo1	133.2 (4)	O1 ^{viii} —Mo1—Mo1 ^{xix}	52.67 (9)

Mo1 ⁱ —Li1—Mo1	116.94 (6)	O3—Mo1—Mo1 ^{xix}	141.60 (11)
Mo1 ⁱⁱⁱ —Li1—Mo1	116.94 (6)	O2—Mo1—Mo1 ^{xix}	94.37 (13)
Mo1 ⁱⁱ —Li1—Mo1	175.1 (5)	O2 ^v —Mo1—Mo1 ^{xix}	139.30 (11)
Mo1 ^{iv} —Li1—Mo1	59.0 (4)	Mo1 ^{xviii} —Mo1—Mo1 ^{xix}	60.0
Mo1 ^v —Li1—Mo1	59.0 (4)	O4 ^{xvii} —Mo1—Li1 ^{xx}	85.0 (3)
O3—Li1—Li2	74.6 (5)	O1 ^{xvii} —Mo1—Li1 ^{xx}	139.97 (14)
O2 ⁱ —Li1—Li2	74.9 (3)	O1 ^{xviii} —Mo1—Li1 ^{xx}	139.97 (15)
O2 ⁱⁱ —Li1—Li2	162.6 (10)	O3—Mo1—Li1 ^{xx}	75.6 (3)
O2 ⁱⁱⁱ —Li1—Li2	74.9 (3)	O2—Mo1—Li1 ^{xx}	43.02 (16)
Mo1 ⁱ —Li1—Li2	120.8 (5)	O2 ^v —Mo1—Li1 ^{xx}	43.02 (16)
Mo1 ⁱⁱⁱ —Li1—Li2	65.8 (4)	Mo1 ^{xviii} —Mo1—Li1 ^{xx}	123.5 (2)
Mo1 ⁱⁱ —Li1—Li2	120.8 (5)	Mo1 ^{xix} —Mo1—Li1 ^{xx}	123.5 (2)
Mo1 ^{iv} —Li1—Li2	60.5 (4)	O4 ^{xvii} —Mo1—Li1	169.2 (3)
Mo1 ^v —Li1—Li2	109.2 (7)	O1 ^{xvii} —Mo1—Li1	67.23 (18)
Mo1—Li1—Li2	60.5 (4)	O1 ^{xviii} —Mo1—Li1	67.23 (18)
O3—Li1—Li2 ^{vi}	74.6 (5)	O3—Mo1—Li1	30.2 (3)
O2 ⁱ —Li1—Li2 ^{vi}	162.6 (10)	O2—Mo1—Li1	100.31 (19)
O2 ⁱⁱ —Li1—Li2 ^{vi}	74.9 (3)	O2 ^v —Mo1—Li1	100.31 (19)
O2 ⁱⁱⁱ —Li1—Li2 ^{vi}	74.9 (3)	Mo1 ^{xviii} —Mo1—Li1	119.49 (18)
Mo1 ⁱ —Li1—Li2 ^{vi}	120.8 (5)	Mo1 ^{xix} —Mo1—Li1	119.49 (18)
Mo1 ⁱⁱⁱ —Li1—Li2 ^{vi}	120.8 (5)	Li1 ^{xx} —Mo1—Li1	105.78 (5)
Mo1 ⁱⁱ —Li1—Li2 ^{vi}	65.8 (4)	Li2 ^{xxi} —O1—Ge1 ^{xxii}	124.9 (8)
Mo1 ^{iv} —Li1—Li2 ^{vi}	60.5 (4)	Li2 ^{xxi} —O1—Mo1 ^{xxiii}	119.4 (6)
Mo1 ^v —Li1—Li2 ^{vi}	60.5 (4)	Ge1 ^{xxii} —O1—Mo1 ^{xxiii}	103.46 (15)
Mo1—Li1—Li2 ^{vi}	109.2 (7)	Li2 ^{xxi} —O1—Mo1 ^{xxiv}	119.4 (6)
Li2—Li1—Li2 ^{vi}	113.2 (5)	Ge1 ^{xxii} —O1—Mo1 ^{xxiv}	103.46 (15)
O4—Li2—O1 ^{vii}	101.1 (7)	Mo1 ^{xxiii} —O1—Mo1 ^{xxiv}	74.66 (17)
O4—Li2—O1 ^{viii}	101.1 (7)	Ge1—O2—Li1 ^{xx}	116.3 (6)
O1 ^{vii} —Li2—O1 ^{viii}	116.4 (5)	Ge1—O2—Mo1	125.62 (15)
O4—Li2—O1 ^{ix}	101.1 (7)	Li1 ^{xx} —O2—Mo1	90.3 (4)
O1 ^{vii} —Li2—O1 ^{ix}	116.4 (5)	Ge1—O2—Mo1 ^{iv}	125.62 (15)
O1 ^{viii} —Li2—O1 ^{ix}	116.4 (5)	Li1 ^{xx} —O2—Mo1 ^{iv}	90.3 (4)
O4—Li2—Li1	74.6 (5)	Mo1—O2—Mo1 ^{iv}	98.6 (2)
O1 ^{vii} —Li2—Li1	65.0 (2)	Li1—O3—Mo1 ^{iv}	115.18 (19)
O1 ^{viii} —Li2—Li1	65.0 (2)	Li1—O3—Mo1	115.18 (19)
O1 ^{ix} —Li2—Li1	175.6 (12)	Mo1 ^{iv} —O3—Mo1	103.2 (2)
O4—Li2—Li1 ^x	74.6 (5)	Li1—O3—Mo1 ^v	115.18 (19)
O1 ^{vii} —Li2—Li1 ^x	175.6 (12)	Mo1 ^{iv} —O3—Mo1 ^v	103.2 (2)
O1 ^{viii} —Li2—Li1 ^x	65.0 (2)	Mo1—O3—Mo1 ^v	103.2 (2)
O1 ^{ix} —Li2—Li1 ^x	65.0 (2)	Li2—O4—Mo1 ⁱⁱⁱ	134.58 (16)
Li1—Li2—Li1 ^x	113.2 (5)	Li2—O4—Mo1 ^{xxiv}	134.58 (16)
O4—Li2—Li1 ^{xi}	74.6 (5)	Mo1 ⁱⁱⁱ —O4—Mo1 ^{xxiv}	76.2 (2)
O1 ^{vii} —Li2—Li1 ^{xi}	65.0 (2)	Li2—O4—Mo1 ^{xxiii}	134.58 (16)
O1 ^{viii} —Li2—Li1 ^{xi}	175.6 (12)	Mo1 ⁱⁱⁱ —O4—Mo1 ^{xxiii}	76.2 (2)

O1 ^{ix} —Li2—Li1 ^{xi}	65.0 (2)	Mo1 ^{xxiv} —O4—Mo1 ^{xxiii}	76.2 (2)
Li1—Li2—Li1 ^{xi}	113.2 (5)		

Symmetry codes: (i) $x-y+1, x, z+1/2$; (ii) $-x+2, -y+2, z+1/2$; (iii) $y, -x+y+1, z+1/2$; (iv) $-x+y+1, -x+2, z$; (v) $-y+2, x-y+1, z$; (vi) $x-1, y, z$; (vii) $x-y, x-1, z-1/2$; (viii) $y+1, -x+y+2, z-1/2$; (ix) $-x+3, -y+1, z-1/2$; (x) $x+1, y, z$; (xi) $x, y-1, z$; (xii) $-x+y+2, -x+2, z$; (xiii) $-y+2, x-y, z$; (xiv) $-x+y+2, -x+2, z-1$; (xv) $-y+2, x-y, z-1$; (xvi) $x, y, z-1$; (xvii) $-x+3, -y+2, z-1/2$; (xviii) $-x+y+2, -x+3, z$; (xix) $-y+3, x-y+1, z$; (xx) $-x+2, -y+2, z-1/2$; (xxi) $-x+3, -y+1, z+1/2$; (xxii) $x, y, z+1$; (xxiii) $x-y+1, x-1, z+1/2$; (xxiv) $-x+3, -y+2, z+1/2$.